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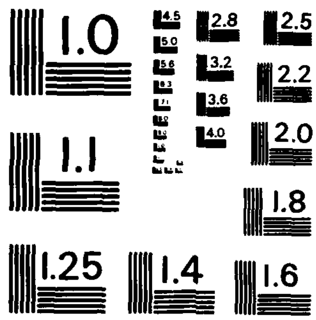

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IN-SITU FORMATION OF A GRAIN BOUNDARY IN FIELD ION MICROSCOPY USING A LASER

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Successful formation and real-time observation is reported of a grain boundary within a view of a FI image using laser beam irradiation in the presence of a high electric field. Formation of the grain boundary in a FI tip appears to be by solid-phase recrystallization due to moderate local heating and extremely high mechanical stresses caused by the electric field and laser irradiation.

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IN-SITU FORMATION OF A GRAIN BOUNDARY IN FIELD ION MICROSCOPY USING A LASER.

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Abstract

Successful formation and real-time observation is reported of a grain boundary within a view of a FI image using laser beam irradiation in the presence of a high electric field. Formation of the grain boundary in a FI tip appears to be by solid-phase recrystallization due to moderate local heating and extremely high mechanical stresses caused by the electric field and laser irradiation.

It is now well established that grain boundaries play essential roles in a wide range of basic kinetic phenomena in polycrystalline materials.[1] Problems such as intergranular corrosion, segregation, recrystallization texture and boundary migration or diffusion rates, are all intimately connected to the nature of the atomic configurations across a grain boundary. Some success in treating these phenomena has been achieved by theoretical models of boundary structure.[2] However, none of the models are able to describe the properties of a monolayer or two on either side of the boundary plane.

The study of grain boundaries is, therefore, best accomplished by experimental methods which provide a direct image of local atomic arrangements across the boundary. Although it is true that some of this information may be obtained by indirect methods, such as diffraction, there are still considerable problems in interpretation, particularly of the ambiguities present in diffuse scattering from non-periodic defects.[3] In this context, TEM (atomic resolution TEM in particular)[4] and FIM are extremely useful and the only techniques capable of direct imaging of grain boundaries.

In spite of this fact, FIM has not found widespread use in the study of grain boundaries. One of the main reasons for this is that it is extremely rare to find a grain boundary in the field of a FI imaging view which encompasses only a small area, not more than 10^{-10} cm^2. All previous FIM studies of grain boundaries utilize the accidentally-found grain boundary.[5] The suggestions made by Wolf[6] and by Mueller[7] have never been followed seriously. Recently, we have conducted a feasibility test on Wolf's treatment, that is, applying cold work to a wire prior to etching it into a tip. Our finding was unsatisfactory. This treatment is only sometimes effective in producing a grain boundary upon annealing. In this paper we report the first successful in-situ grain boundary formation in a FIM using laser irradiation. The system used is a conventional field ion microscope which is discussed in detail elsewhere.[8] A Nd-YAG laser of λ = 10600 Å (1 mm^2 beam size, 100 mJ/10 ns pulsed laser) was used. A laser beam was introduced to the chamber through a 6" OD pyrex glass view-port attached to the side wall of the chamber and irradiated a FI tip. A set of lenses mounted on a micrometer and neutral filters were used to control the beam position and beam intensity. In the present experiment the second harmonics of 5300 Å green light was used for safety and easy focusing. The laser power was varied in the range of 0 to 0.5 mJ/pulse.

The experimental procedure is as follows. A FI tip is [illegible]

~25K using a closed-cycle helium liquefier with the base pressure of $10^{-[illegible]}$ Torr. In the presence of an imaging gas ($\sim 10^{-4}$ Torr) the tip voltage is applied and surface layers are field evaporated until a surface becomes a smooth hemi-spherical shape. Then, the tip voltage is lowered to produce the best image. Laser pulses are now applied to the tip. Using a relatively small power (0.1 mJ) the position of the laser beam is brought into the cap of the tip. When the beam position is adjusted to be right at the cap of the tip, one observes orderly evaporation of the surface atoms, known as laser-enhanced field evaporation.[9] Then, the laser power is increased gradually to the desired value while the tip voltage is decreased to maintain the surface evaporation at a nominal rate (approximately a few layers per sec). Evaporation by the laser in the presence of the electric field appeared to be well-controlled. Laser irradiation appears to make a tip blunt much faster than does the ordinary field evaporation. Thus, the tip voltage was increased gradually to keep the effective field constant. The moment at which a grain boundary forms can not usually be predicted except for a few cases where minute changes are first observed and the formation of a grain boundary follows a moment later.[10] Therefore, the laser irradiation was continued until gross changes were observed in a FI image while gradually increasing the tip voltage. The whole sequence was video-monitored. Still photos of the FI images were also taken. When a grain boundary formed, the laser irradiation was immediately stopped. The tip voltage was increased to remove surface irregularities until a smooth well-defined image was obtained. When damage appeared to be very extensive, a tip was annealed in-situ by Joule-heating. This in-situ annealing is very effective to restore a well ordered surface, although a tip radius inevitably increases.

Fig. 1 shows a typical example of grain boundary formation by this method. A good FI image of a tungsten tip with the [110] axis was obtained at 5.35kV and 25K (Fig.1(a)). This tip was exposed to the laser (10Hz) at the power [illegible]

0.4mJ/pulse). The surface layers evaporated rapidly and the tip voltage was increased intermittently to maintain a constant evaporation rate. A grain boundary was formed after 10 sec of laser irridation when the tip voltage was raised to 6.80 kV. The surface showed some irregularities at the termination of laser irradiation. The tip voltage was increased to 8.69 kV for field evaporation and a FI image (Fig. 1(b)) was taken at the best image voltage (7.877kV) in which one can observe a sharp grain boundary extending from 12 to 7 o'clock. The right side of the grain boundary has essentially the same crystallographic orientation as the original material although the radius is larger at 570 Å than the original 320 Å. Fig. 1(c) is a stereographic projection of the surface in Fig. 1 (b). It is evident from this map that the boundary is a twin boundary with respect to the [211] axis, which is also described as = 3 with a rotation of 71° around the [110] axis. Further evaporation of the surface layers did not change the geometrical structure of this surface, suggesting that the grain boundary plane is essentially parallel to the [110] axis. Fig. 2 shows another example of in-situ grain boundary formaton by laser irradiation. Laser irradation (1 to 10 Hz and 0.3 to 0.5 mJ/pulse) was administered for approximately 40 min to a well-defined clean W tip imaged initially at 3.73 kV (Fig. 2(a)), when a grain boundary was formed at the upper left corner. After terminating laser irradiation, the surface was field evaporated to show a clean surface, Fig. 2(b). A grain boundary runs from 12 o'clock to 8 o'clock. An additional 13 layers of field evaporation resulted in the micrograph, Fig. 2(c), imaged at 4.602 kV. Fig. 2(d) was obtained by field evaporation of 60 more atomic layers. The grain boundary b-d appears to move toward the center of the tip, the (110) plane. This is due to the fact that the boundary plane is not parallel but inclined to the tip axis. A similar mapping of this surface indicated that this grain boundary is likely to be = 17 with a rotation of 87° around the [110] axis.

We were able to create a grain boundary in 14 out of 24 tips tried so far. Four others exhibited minor defects, such as screw dislocations, vacancy clusters and slip bands but no grain boundary to the end of the experiment. The rest of the tips (six) were damaged so extensively by laser irradiation that image could not be obtained thereafter. Our success rate of over 58 % is surprisingly good and it is fair to say without reservation that a grain boundary can be created within a view of a FI image in a well-controlled fashion.

In order to understand the mechanism of the grain boundary formation in a FI tip, we evaluated the surface temperature rise during laser irradiation. The method used here is based on the experimental fact that the evaporation field at a fixed evaporation rate decreases as the surface temperature increases and is a unique function of temperature.[11] The evaporation field was measured while varing the laser power in the range 0 to 0.5 mJ/pulse and was converted to the surface temperature. The result is shown in Fig. 3. The surface temperature increased roughly 400 degrees during laser irradiation at the maximum power in the present experiment. The surface temperature rise was estimated based on the thermal conduction model[12] that the surface is radiated by a heat pulse f_0 for the first τ sec:

$$-K\frac{\partial T}{\partial x} = f_0 \qquad 0<t<\tau$$

$$= 0 \qquad \tau<t$$

The solution is given as

$$T = T_0 + \sqrt{\kappa t}\,\frac{2f_0}{K}\,\mathrm{ierfc}\,(x/2\sqrt{\kappa t}) \qquad 0<t<\tau$$

$$= T_0 + \sqrt{\kappa t}\,\frac{2f_0}{K}\,\mathrm{ierfc}\,(x/2\sqrt{\kappa t}) - \sqrt{\kappa(t-\tau)}\,\frac{2f_0}{K}\,\mathrm{ierfc}(x/2\sqrt{\kappa(t-\tau)})$$

$$\tau<t$$

The maximum temperature rise at the surface is

$$\Delta T = T(x=0,\ t=\tau) - T_0$$

$$= \frac{2f_0}{K}\sqrt{\kappa\tau/\pi}$$

We have obtained $\Delta T = 420^{\circ}C$ for $E_0 \approx 0.5$ mJ/pulse and $\Delta T = 1200^{\circ}C$ for $E_0 =$ 2 mJ/pulse in the case of a tungsten tip, in good agreement with the measured surface temperature increase. This rules out the possibility of liquid Phase recrystallization in the formation of a grain boundary by this method.[13] The formation of a grain boundary in the present work is, therefore, seemingly by solid phase recrystallization due to the presence of extremely high stress caused by the high electric field and assisted by a laser heat pulse. In conclusion, we have succeeded for the first time in creating and observing a grain boundary within a view of a FI image in-situ using a laser pulse. This is truly a breakthrough in the FIM study of grain boundaries and segregation at grain boundaries.

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REFERENCES

1. R. W. Balluffi (ed.), Grain-Boundary Structure and Kinetics, American Society of Metals, Metal Park, Ohio 1980.
2. W. Bollmann, Crystal Defects and Crystalline Interfaces, Springer-Verlag, New York, N.Y. 1970.
3. W. Gaidig and S. L. Sass, Phil. Mag. 39A, 725 (1979).
4. G. Thomas and M.J. Goringe, Transmission Electron Microscopy of Materials, J. Wiley-Interscience, New York, N.Y. 1979.
5. B.Loberg and H. Norden, pl, Grain Boundary Structure and Properties, G. A. Chadwick and D. A. Smith (eds.), Academic Press, London 1976.
6. P. Worf, Abstract of the 6th Field Emission Symposium, Washin[illegible] D.C.. (1959)
7. E. W. Mueller, Proceedings of IV International Kongress f. Elek[illegible]onen Mikroskopie, Berlin 1958,Vol.1 820 (Springer-Verlag. Berlin 1960).
8. A. Jimbo and T. Sakurai, to be published.
9. G. L. Kellogg, J. Appl. Phys. 51, 1184 (1980)
10. Detailed analysis of video-taped real-time image changes are currently attempted and to be published.
11. S.Nakamura and T. Kuroda, Surf. Sci. 17, L346 (1969)
12. H. S. Carslaw and J. G. Jaeger, Conduction of Heat in Solids, Oxford Press,Oxford 1959.
13. M. O. Thompson, J. W. Mayer, A. G. Cullis, H. C. Webber, N. G. Chew, J. M. Poate and D. C. Jacobson, Phys. Rev. Lett. 50, 896 (1983), and J.F.Young, J.S.Preston, N. M. van Driel and J. E. Sipe, Phys. Rev. B27, 1155 (1983)

FIGURE CAPTIONS

Figure 1(a). A micrograph of clean tungsten prior to laser irradiation taken at $V_{tip} = 5.35$ kV.

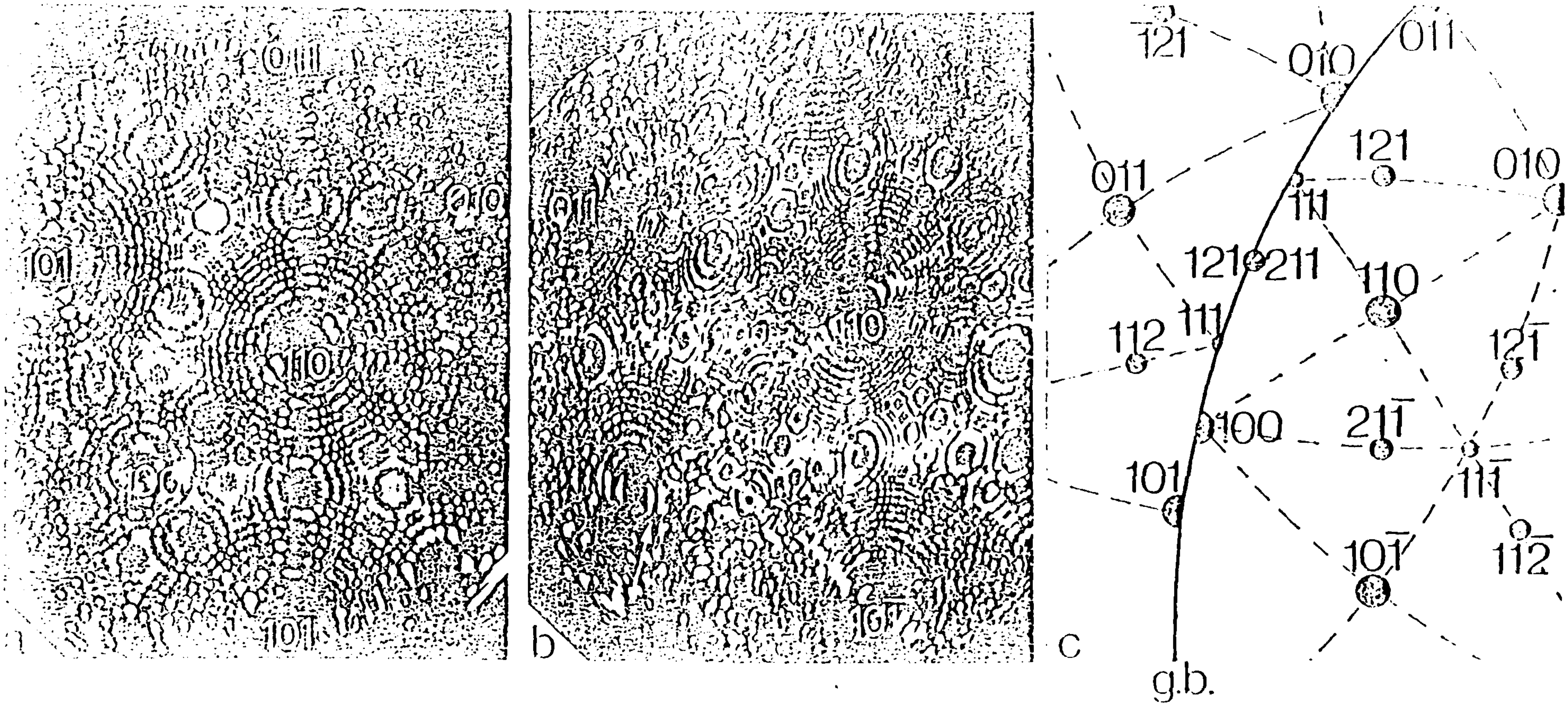
(b). A grain boundary $\Sigma=3$ with a rotaion of 71° around the [110] axis was created by laser irradiation. The micrograph was taken at $V_{tip} = 7.877$ kV upon field evaporation at 8.695 kV.

(c). Stereographic map of micrograph (b).

Figure 2(a). A micrograph of clean tungsten taken at $V_{tip} = 3.73$ kV. Laser irradiation produced a grain boundary $\Sigma=17$ with a rotation of 37° around the [100] axis.

(b) to (d). Further field evaporation of the surface layers resulted in the apparent movement of the grain boundary toward the center of the tip.

Figure 3. The surface temperature rise (ΔT) as a function of the laser power.



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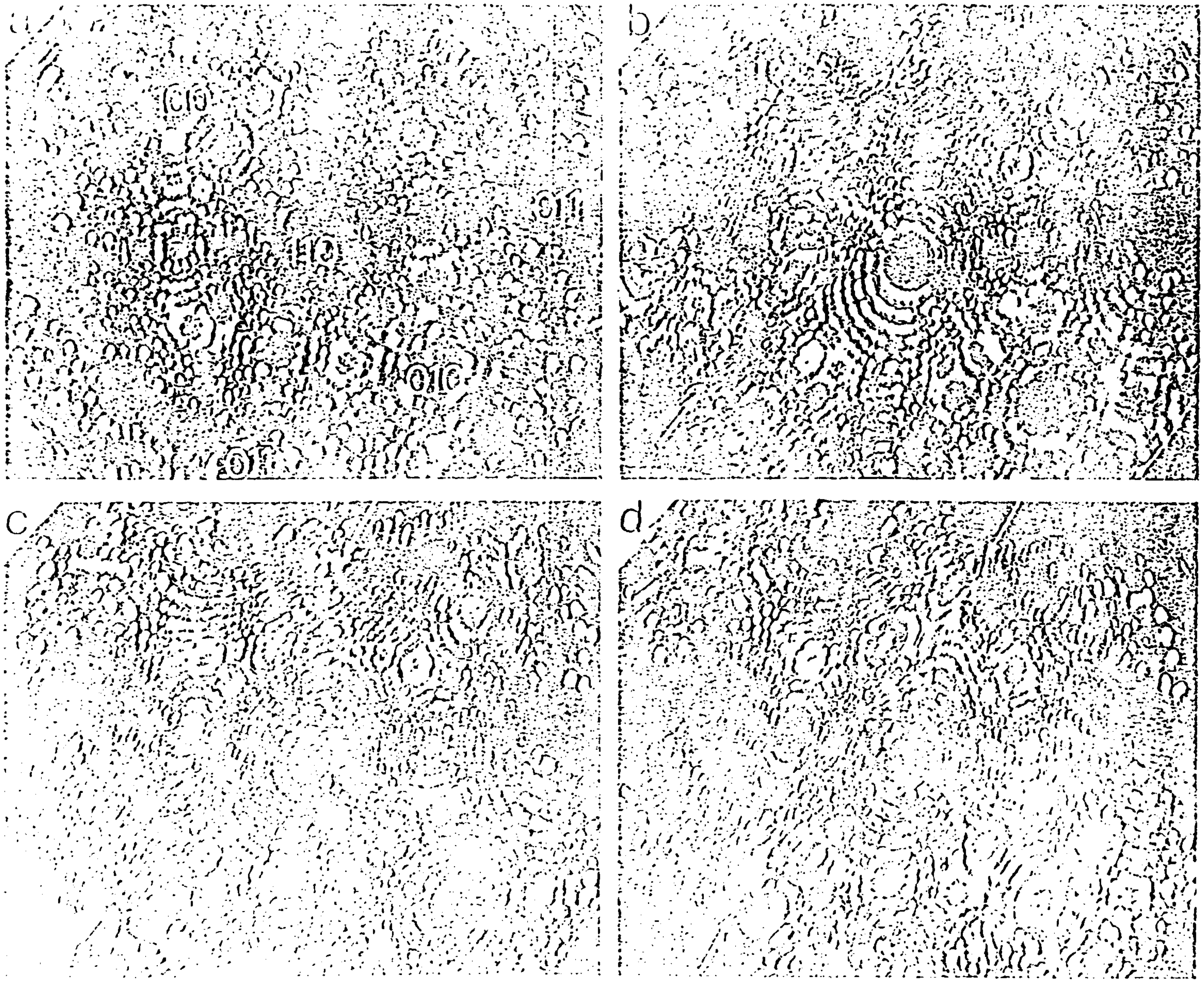

Fig.2. T. Sch[illegible]
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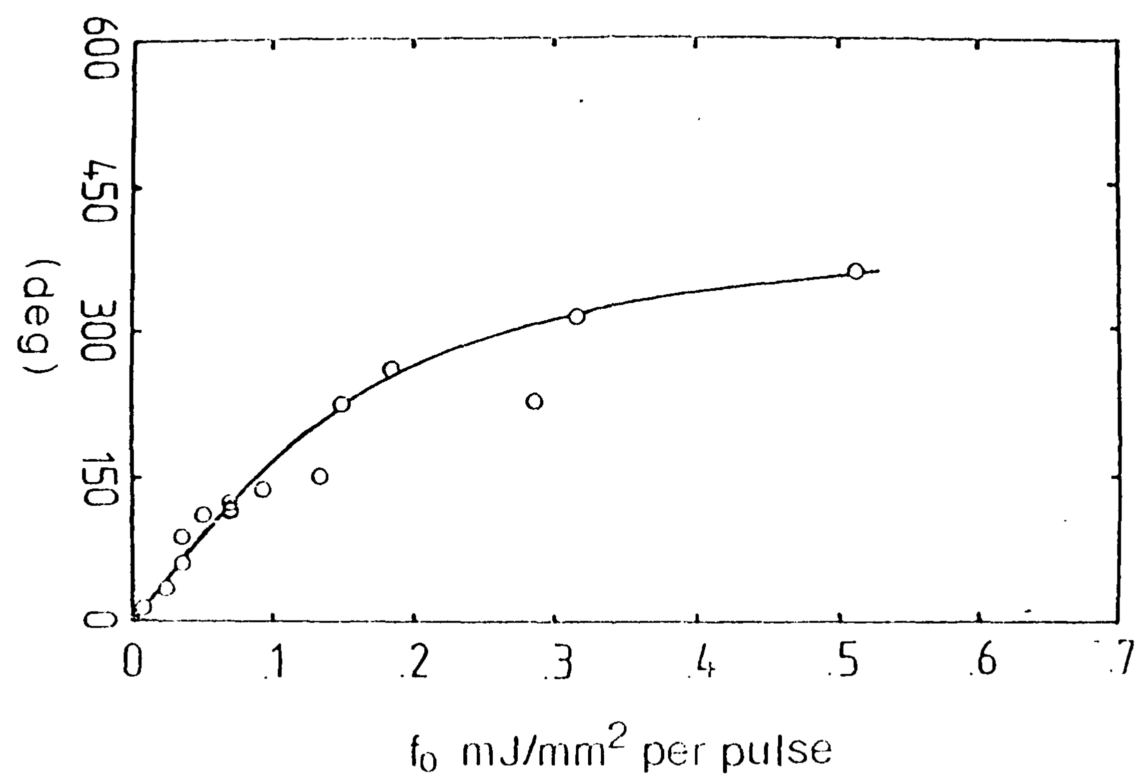

Fig.3. T. Sakurai
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